

ELECTRICAL CHARACTERIZATION OF SiGe THIN FILMS

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ABSTRACT

An apparatus for measuring electrical resistivity and Hall coefficient on both thin films and bulk material over a temperature range of 300K to 1300K has been built. A unique alumina fixture, with four molybdenum probes, allows arbitrarily shaped samples, up to 2.5 cm diameter, to be measured using van der Pauw's method. The system is fully automated and is constructed with commercially available components. Measurements of the electrical properties of doped and undoped Si-Ge thin films, grown by liquid phase epitaxy reported here, are to illustrate the capabilities of the apparatus.

INTRODUCTION

Improvement of the high temperature thermoelectric conversion efficiency of hot-pressed n-type Si-Ge alloys required that substantial increase in the carrier concentration of these materials be made [1]. Because of the limited solid solubility of the dopants used, combinations of several elements such as Ga and P have been used [2,3]. However, the understanding of the doping mechanisms in the hot-pressed materials is quite complex and the results are difficult to reproduce. To better investigate the behavior of these alloys, characterization of homogeneous single crystalline materials are needed. For this purpose, growth of high quality Si-Ge thin films doped with various elements has been carried out using liquid phase epitaxy (LPE), a near-thermodynamic equilibrium process [4].

Characterizing these thermoelectric materials also involved measuring their electrical transport properties, using a Hall effect apparatus. Because of the significant effects of dopant precipitation on the properties and of the high temperature range of interest, measurements needed to be conducted from room temperature up to 1000 °C.

A common method for measuring resistivity and Hall coefficient is based on van der Pauw's technique [5]. This technique allows for flat arbitrarily shaped discs to be tested, provided that the electrical contacts are sufficiently small and located along the circumference of the sample, and the sample is free of (geometrical) holes.

From these measurements, the following properties can be obtained: electrical resistivity, Hall coefficient, Hall mobility, and Hall concentration. Due to the high temperature range of applications of the Si-Ge thermoelectric alloys, the fixture design, its implementation, and the measuring process presented a variety of problems over standard room temperature measurements such as choice of materials, electrical connections, measurement and temperature control.

The system is built with commercially available equipment and is fully automated with an AT compatible computer, via an IEEE-488 interface. The software is written in Microsoft's Quick Basic 4.5 for its fast prototyping capabilities and ease of use.

APPARATUS DESIGN

Because of the variety of sample shapes and sizes to be tested, van der Pauw's method was chosen for making both Hall effect and resistivity measurements. This method offers great flexibility and ease of implementation, including the inherent advantages of (i) accepting planar samples of arbitrary shape, and thickness and (ii) avoiding electrical problems associated with misalignment of the Hall probes in the conventional Hall-bar configuration.

We use a DC technique for our Hall measurements because it simplifies the electronic instrumentation. It has the disadvantages of reduced sensitivity and emf noise, both thermal and spurious. In the DC technique thermal emfs are cancelled by reversing the current for each resistance measurement. Spurious emfs are greatly reduced by averaging techniques.

The design and construction of a high temperature Hall effect apparatus is problematic, due to the temperature constraints in the choice of materials, the inability to maintain electrical contact integrity, and the confined space between the magnet poles.

The fixture body on figure 1 is made of alumina because of its strength and electrical insulation at high temperatures. Molybdenum is used for the electrical circuit for its machining and welding ease over tungsten.

The molybdenum probes apply pressure to the sample, assuring that electrical point contact is maintained throughout the temperature cycle.

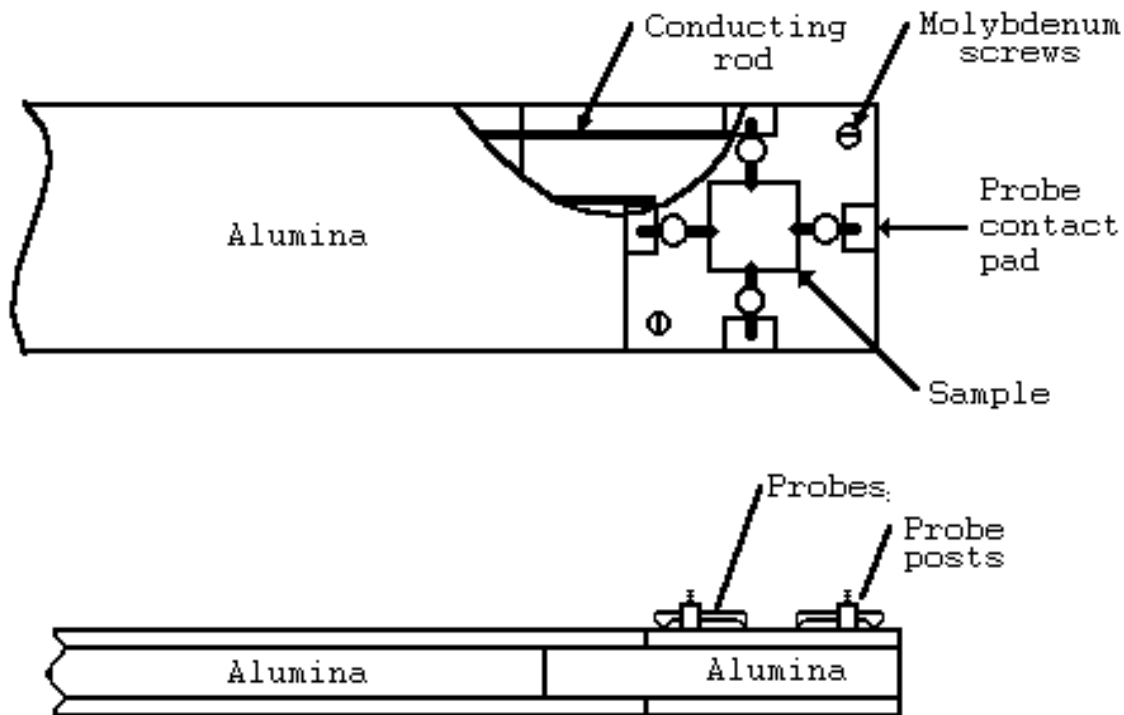


FIG.1 Hall effect fixture

This has presented problems on the cooling part of the temperature cycle due to the probes not springing back, causing a loss of electrical contact. To address this problem a tungsten piece is placed on top of the probe to distribute the load of the pressure screw.

The fixture is only 2.54 cm thick fitting in the preexisting furnace and vacuum chamber located between the poles of the electromagnet [6]. It has been designed to hold two samples for measurement.

A block diagram of the electronic instrumentation is presented in figure 2. All equipment is IEEE 488 addressable and commercially available. The system consists of the following equipment:

- furnace power supply (HP 6030A)
- electromagnet (Harvey-Wells Corp. L-128)
- magnet DC power supply (Sorensen DCR150-70)
- A/D magnet power supply controller (Sorensen 488MicroDap)
- Gaussmeter (Magnetic Instrumentation Inc. 7305-IEEE)
- Keithley 706 scanner
- Keithley 220 current source
- Keithley 180 nanovoltmeter
- Keithley 7168 nanovolt scanner card
- Keithley 7065 Hall effect cards
- vacuum gauge (Leybold Inficon Inc.. CC3)
- turbo pump (Leybold Heraeus Turbovac 360 CSV)
- vane pump (Leybold Heraeus Trivac D8B)
- AT IBM-compatible computer
- CEC IEEE 488 interface bus

The system is fully automated and the software is written in Microsoft's Quick Basic 4.5. All test parameters are programmable, and some, like the current output and voltmeter range, are self adjusting to optimize resolution.

We use guarded triaxial cable for all external electrical wiring to the sample and from the current source. Connections are made using low thermo-emf solder to improve on the signal to noise ratio.

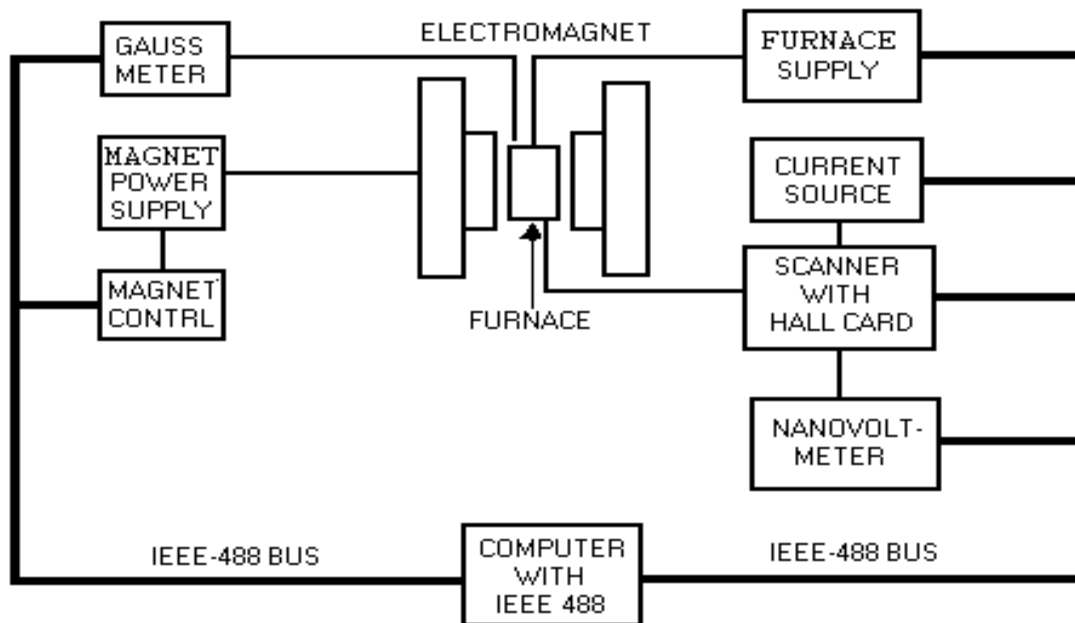


FIG. 2 ELECTRONIC INSTRUMENTATION DIAGRAM

EXPERIMENTAL PROCEDURE

Once we have verified that the lamellar specimen conforms to van der Pauw's specifications, we can proceed with the measurement by mounting the sample to the fixture and securing it with the molybdenum probes. Since the apparatus is completely automated, the operator has only to enter the sample characteristics and assign a file name for recording the results.

Throughout the temperature cycle the following measurements are repeated continuously, taking an average of 11 minutes for a complete measurement. The temperature usually shifts about 12 °C from the beginning to the end of the measurement. Most tests consist of over 100 data points for the full temperature cycle.

An electrical integrity test is performed on the system to check the probe-to-sample contact. This simple ohmic test is done by passing current through two adjacent contacts and measuring the voltage drop on the same pair of leads.

Resistances are measured for currents in both directions and averaged to cancel thermal emfs. Both low voltages (8 to 10 V typically) and currents (100 mA for SiGe) are used to minimize current induced thermal gradients on the sample.

The resistivity is calculated using van der Pauw's four probe method. Resistances are measured by the above method and the resistance-ratio function (f) is calculated.

Hall coefficient is calculated by averaging the Hall resistances for the magnetic field in both directions and having canceled thermal emfs.

Measurements consist of a running window average, where seven resistances are averaged, and the standard deviation used as an acceptance criteria. If the criteria is not met, another measurement is added to a fifty element array and reevaluated until the condition is met or the array is full. Once the array is full it is averaged, this avoids staying too long at any given temperature, and allows for a tighter acceptance criteria.

All of the data is saved on disk in ASCII format so it can be used with available commercial software packages, such as Jandel's SigmaPlot.

RESULTS AND DISCUSSION

The doped thin film samples tested were deposited by liquid phase epitaxy (LPE) on a highly resistive B-doped (111)-oriented Si substrate. Dopants included Ga, Al, Sn and P. Typically, the thickness of the Si substrate is 360 μm while the thickness of the Si-Ge layers ranges from 10 to a maximum of 30 μm . Although the electrical resistivity of the Si substrate was no less than 300 times larger than any LPE sample at room temperature, this gap was expected to decrease significantly at high temperatures. For that reason, a plain substrate was also measured up

to a 1000 °C in order to demonstrate that there was little or no interference during the measurement of the thin films. The electrical resistivity of several LPE samples was compared to the Si substrate resistivity for the entire temperature range and plotted on figure 3.

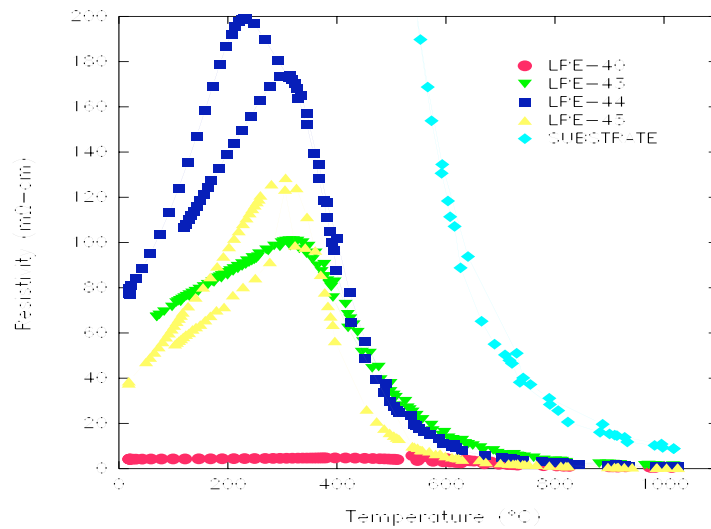


Figure 3: Electrical resistivity versus temperature of a Si substrate and several LPE samples. [Axes, labels missing in this author manuscript]

At about a 1000 °C, the resistivity of the Si substrate dropped to values only ten to thirty times larger than the SiGe thin films (around 8.5 mΩ.cm). While this resistivity ratio substrate to sample is comfortable enough to ensure a meaningful measurement of the properties of the films, readings of the very small values of the Hall voltage became noisy for the LPE samples with the highest resistivities (LPE-43 and LPE-44) attributed to some substrate interference. Measurements of the more heavily doped films were not appreciably affected.

All the samples measured in this work changed from p-type to n-type conductivity in a 300-400 °C temperature range because of low doping levels (LPE-40 and LPE-43), or of the changes of the solid solubility limits of Ga and P present in solution [4] with temperature (LPE-44 and LPE-45). Figure 4 presents the Hall concentration calculated from the Hall coefficient for the whole temperature range. On figure 5, Hall mobilities of less than $1 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ have been measured when the Si-Ge thin film conductivity type is at a transitional stage.

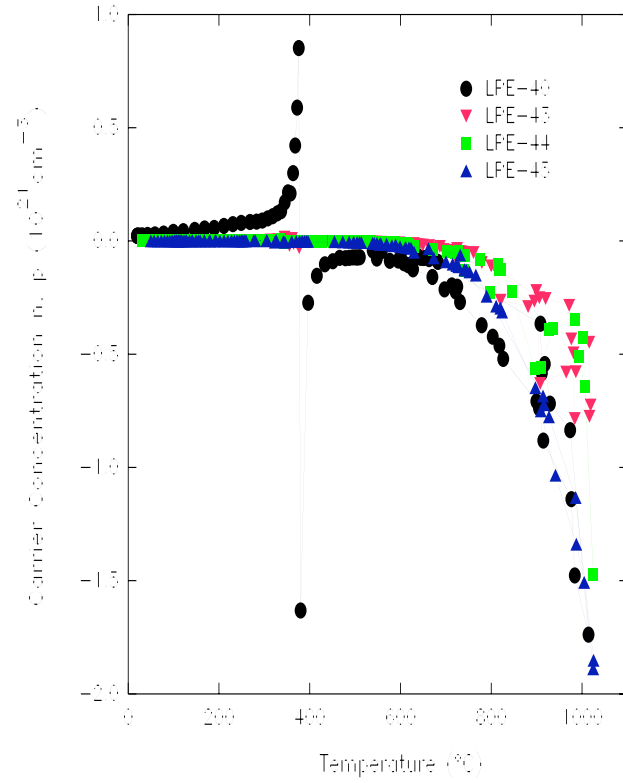


Figure 4: Hall concentration versus temperature for various doped Si-Ge thin films. A change in conductivity type occurred at 375°C. [Axes, labels missing in this author manuscript]

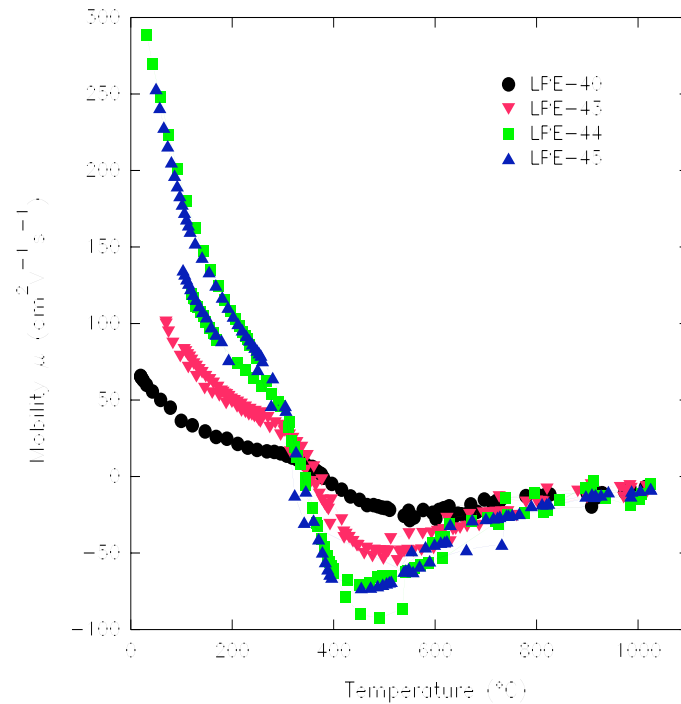


Figure 5: Hall mobility versus temperature for various doped Si-Ge thin films. [Axes, labels missing in this author manuscript]

The electronic noise for some of these measurements can be greater than 50% of the signal, but most of these measurements still follow the curve's path, indicating that the randomness of the noise cancels with averaging.

Another source of errors in the measurement is discussed in van der Pauw's paper; these are due to contact size and its position with respect to the sample periphery. Since the design of the molybdenum probes ensures point contacts, positioning of the probes to the sample periphery was the only concern. To address this potential source of

error, a series of experiments was conducted, where an operator visually aligned the probes to the sample periphery, and measured the samples several times to find the average error introduced experimentally. This was done on two samples, one a disc, and the other a clover-leaf configuration, a design that substantially reduced the errors [5]. The experiments showed no appreciable differences during the course of the measurements, demonstrating that it is possible to visually align probes close enough to the sample periphery in a reproducible fashion. The calculated relative error on the probe to sample placement resulted in values of less than 1% for the electrical resistivity and less than 5% for the Hall coefficient, the Hall mobility and the Hall concentration.

CONCLUSION

A new station capable of conducting high temperature Hall effect and electrical resistivity measurements has been built. Its novel fixture design allows for both, planar bulk samples and well thin films, to be measured from room temperature up to 1000 °C. Reliable measurements and temperature control were achieved by developing new software in an easy-to-use language for operating commercially available IEEE-addressable electronic instrumentation.

Several doped Si-Ge thin films samples, deposited on highly resistive Si substrates, have been measured to demonstrate the capabilities of the new apparatus, contributing to the understanding of these thermoelectric materials. Reproducibility of the probe placement on the sample periphery and quality of the point contacts have been satisfactorily checked.

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